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A PRELIMINARY INVESTIGATION OF THE INTERACTION OF M-50 STEEL WITH A PERFLUOROALKYLETHER FLUID AND A 1 PERCENT ADDITIVE-FLUID FORMULATION

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TECHNICAL REPORT AFML-TR-77-125
Interim Report for Period 1 March 1976 to 1 March 1977

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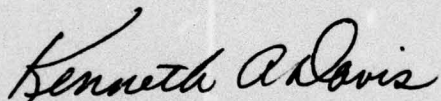When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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LT. COL. KENNETH A. DAVIS
Project Monitor

FOR THE COMMANDER

J.M. KELBLE
Nonmetallic Materials Division
Air Force Materials Laboratory

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Electron spectroscopy for chemical analysis (ESCA) has been used to characterize the passivation film formed on the surface of M-50 steel coupons during O-C testing in a perfluoroalkylether fluid containing a perfluoroalkylether aryl phosphine additive. Prior to analysis of the metal coupons, samples of the additive and fluid which had not undergone O-C testing were characterized with ESCA. The measured atom percent compositions of the fluid and additive were found to agree well with the compositions ascertained

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20. ABSTRACT: (continued)

from the formulae. Freshly polished metal coupons were analyzed
and were found to contain high levels of carbon and oxygen surface
contamination. On coupon which had undergone O-C testing in
additive-fluid formulations, surface phosphorous levels were found
to be roughly five times greater than in the additive alone. This
data suggests that in the protective action afforded during the
O-C test, the additive has changed chemistry.

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FOREWORD

This report was prepared by the University of Dayton Research Institute, Dayton, Ohio under Contract No. F33615-75-C-5095. The work was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, AFSC, with Lt. Col. Kenneth A. Davis, as project monitor.

This report covers work conducted from 1 March 1976 to 1 March 1977.

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SECTION I
INTRODUCTION

Polyperfluoroalkylethers are stable, high temperature fluids, with other physical properties making them suitable for use as gas turbine engine oils, hydraulic fluids, and advanced lubricants. These fluids exhibit, however, metal corrosion effects at high temperatures when in an oxidative environment. Oxidation-corrosion (O-C) testing of these fluids,[1] demonstrates that metal corrosion is not a simple process. There is a strong indication that metal corrosion is caused by oxidation products of the fluids interacting with the metal.

Additives have been developed which inhibit corrosion.[1] It would be helpful to understand the role the additive is playing in inhibiting metal corrosion. Two mechanisms have been proposed. One involves the additive acting simply as a metal deactivator, that is, the additive forms a chemical bond with the metal, producing a protective film. The second involves the additive acting as a free radical trap for fluid degradation products. In this second mechanism the additive is proposed to react with free radicals formed from perfluoro-polymer fluid, thus forming a mixture which is not as reactive with the metal. In this report we have made some preliminary studies on the oxidation inhibiting action of a perfluoroalkylether aryl phosphine additive.

In these studies, electron spectroscopy for chemical analysis (ESCA) was used to probe differences in the chemistry of the additive and the fluid both before and after mixing. ESCA was also used to examine metal coupon surfaces before and after O-C testing.

[1]C.E. Snyder and R.E. Dolle, <u>ASLE Transactions</u>, <u>19</u> <u>(3)</u> 171 (1976).

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1.   O-C TEST AND SAMPLES

Liquid samples, before O-C testing, of the additive, the
fluid (both structures are shown in Figure 1) and a 1 percent
formulation of the additive in the fluid (hereafter called the
formulation) were analyzed in addition to M-50 alloy metal coupon
samples. The metal coupons submitted were subjected to one of
the following conditions prior to analysis: a) pre-test freshly
polished (Appendix A describes the polishing procedure) b) post-
test-(CS225I) stressed at 650°F in the fluid only, c) post-test-
(CS211I) stressed at 625°F in the formulation, and d) post-test-
(CS220I) stressed at 650°F in the formulation. The O-C test, to
which reference is made, is the MANL-micro oxidation-corrosion
test using a reduced (1 liter/hr) air-flow and ferrous alloy
coupons.[1] The O-C results for CS211I, CS220I and CS225I are shown
in Table I. The acceptable limit for metal weight change for
the metal coupons is ± 0.2 mg/cm^2. Table I indicates that the
additive present in tests CS211I and CS220I does inhibit corro-
sion when compared with the test using the fluid alone (CS225I).

TABLE I

O-C TEST DATA FOR SAMPLES SUBMITTED

Sample	Temp (°F)	Δ Viscosity @ 100°F (%)	Fluid Loss (%)	M-50* Metal Wt Changes mg/cm^2
CS211I	625	+1.48	0.33	+0.07
CS220I	650	+0.58	0.60	+0.33
CS225I	650	+0.32	10.18	-4.74

*Metal coupon type of interest in the study although other metals
 were also used in the O-C test.

[1]C.E. Snyder and R.E. Dolle, ASLE Transactions, 19 (3) 171 (1976).

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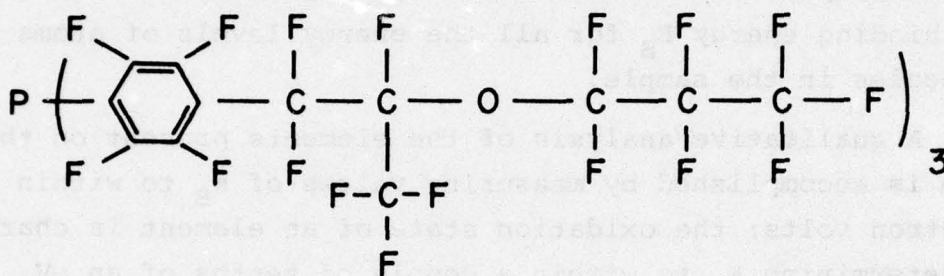
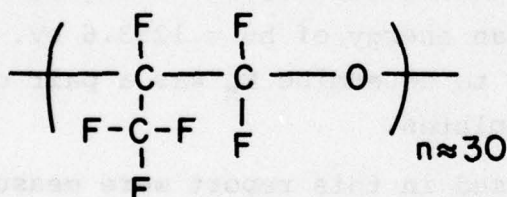**ADDITIVE :**

FLUID (H F P O POLYMER):

Figure 1.   Structures of Additive and Fluid

SECTION II
THE EXPERIMENTAL TECHNIQUE

The analytical technique ESCA involves the interaction of x-ray photons having a constant energy, $h\upsilon$, with bound electrons in a sample, having a binding energy E_B. Via the photoelectric effect, these bound electrons are promoted into a continuum outside the sample as photoelectrons with a kinetic energy, E_K. By measuring the various values of E_K for photoelectrons ejected from a sample, one can determine by simple subtraction ($h\upsilon - E_K$) the binding energy E_B for all the energy levels of atoms or molecules in the sample.

A qualitative analysis of the elements present on the surface is accomplished by measuring values of E_B to within a few electron volts; the oxidation state of an element is characterized by determining E_B to within a couple of tenths of an eV. Semi-quantitative surface atom concentrations are ascertained by measuring the photoelectron peak heights and adjusting for the sensitivity of the instrument to the different elements. The x-ray source used in the following results was a magnesium anode, which has a $K\alpha_{1,2}$ line with an energy of $h\upsilon = 1253.6$ eV. The electron energy decoder used to determine E_K was a pair of hemispherical electrostatic plates.

The ESCA spectra discussed in this report were measured from samples in high vacuum (approximately 1×10^{-7} Torr pressure). For the compounds of interest the technique is nondestructive. The liquid samples studied were dropped onto a stainless steel sample holder such that the liquid wetted the surface evenly. Since ESCA is sensitive to only the top, approximately 10Å of a sample, the stainless steel holder did not interfere with ESCA measurements on the liquids. Also, the vapor pressures of the liquids were sufficiently low so that there was no noticeable evaporation of the sample in the time required for a complete analysis.

SECTION III
ANALYSIS OF KNOWN LIQUID MATERIALS -- ADDITIVE, FLUID, AND MIXTURE

The structures of the additive and the fluid used in this study are presented in Figure 1. Figure 2 presents the overall ESCA scans of a) the additive, b) the fluid, and c) the formulation. Fluorine, oxygen, carbon, and chlorine were found in all three samples. A trace of sulfur was also observed in each sample. Phosphorous was below the detectability limit in these overall scans. In order to study the oxidation states of the elements present in each of the samples, a narrow energy band surrounding the major peak for a given element in the sample was scanned at high resolution. Figure 3 shows the carbon 1s spectra for a) the additive, b) the fluid, and c) the formulation. Each of the peaks is labeled to indicate the chemical environment of the carbon corresponding to each of the particular peaks. Notice that, for example, in the spectrum of the additive, there are four carbon 1s peaks corresponding to $-CF_3$ (or $-CF_2-0-$), $-CF_2-$ (or $-CF-0-$), $-CF-$, and $C-C$ (or $P-C$) type carbons. Table II presents the data obtained from the high resolution scans of the carbon 1s, oxygen 1s, fluorine 1s, and phosphorous 2p peaks for each of the three samples. The measured atomic percent composition for the additive and the fluid agrees with the theoretical atomic percent composition ascertained from the formulas illustrated in Figure 1. These theoretical values are also given in Table II.

In the carbon 1s spectrum for the formulation (see Figure 2), the carbon peak at approximately 288 eV is much greater in intensity that would be expected for a simple mixture of 1 percent additive in fluid. As an empirical method of determining the amount of additive present in an unknown mixture of fluid and additive, the intensity ratio of the carbon peaks at approximately 293 eV and approximately 291 eV was used. Table III gives this ratio $\dfrac{C_{293}}{C_{291}}$ for the additive, the fluid, and the formulation.

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F 2s

P2p (?)

S2p

Cl 2p

C1s (285)
(291)

F1s (Al Kα$_{1,2}$)

O1s (532)

F Auger (597)

F Auger (625)

F1s (686)

BINDING ENERGY

INTENSITY (ARBITRARY UNIT)
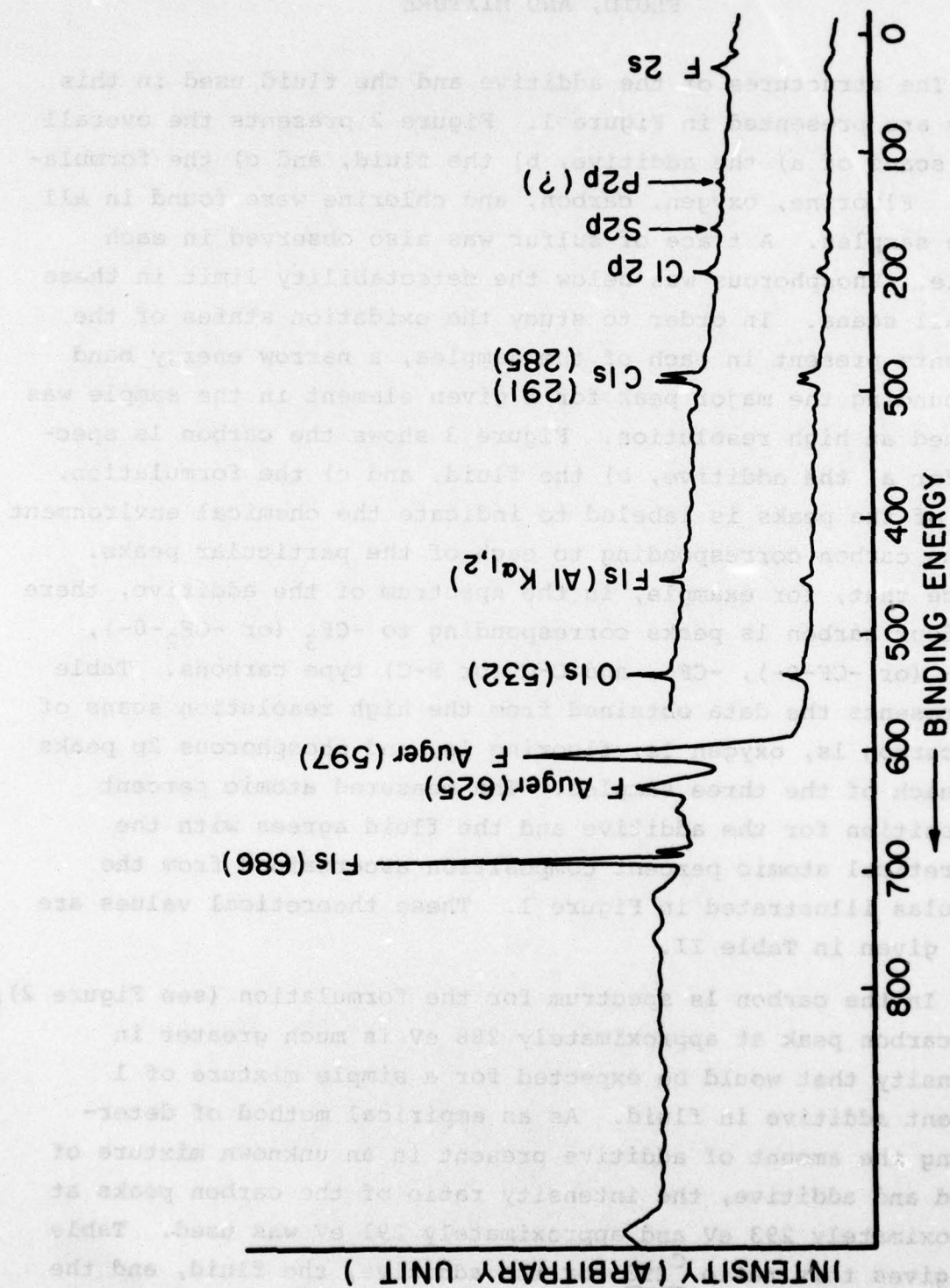
Figure 2. ESCA Overall Spectra of Additive, Fluid, and Formulation.
a. Additive

F2s

P2p (?)

S2p

Cl 2p (199)

C 1s (290)

F 1s (Al Kα$_{1,2}$)

O 1s (532)

F Auger

F Auger

F 1s (686)
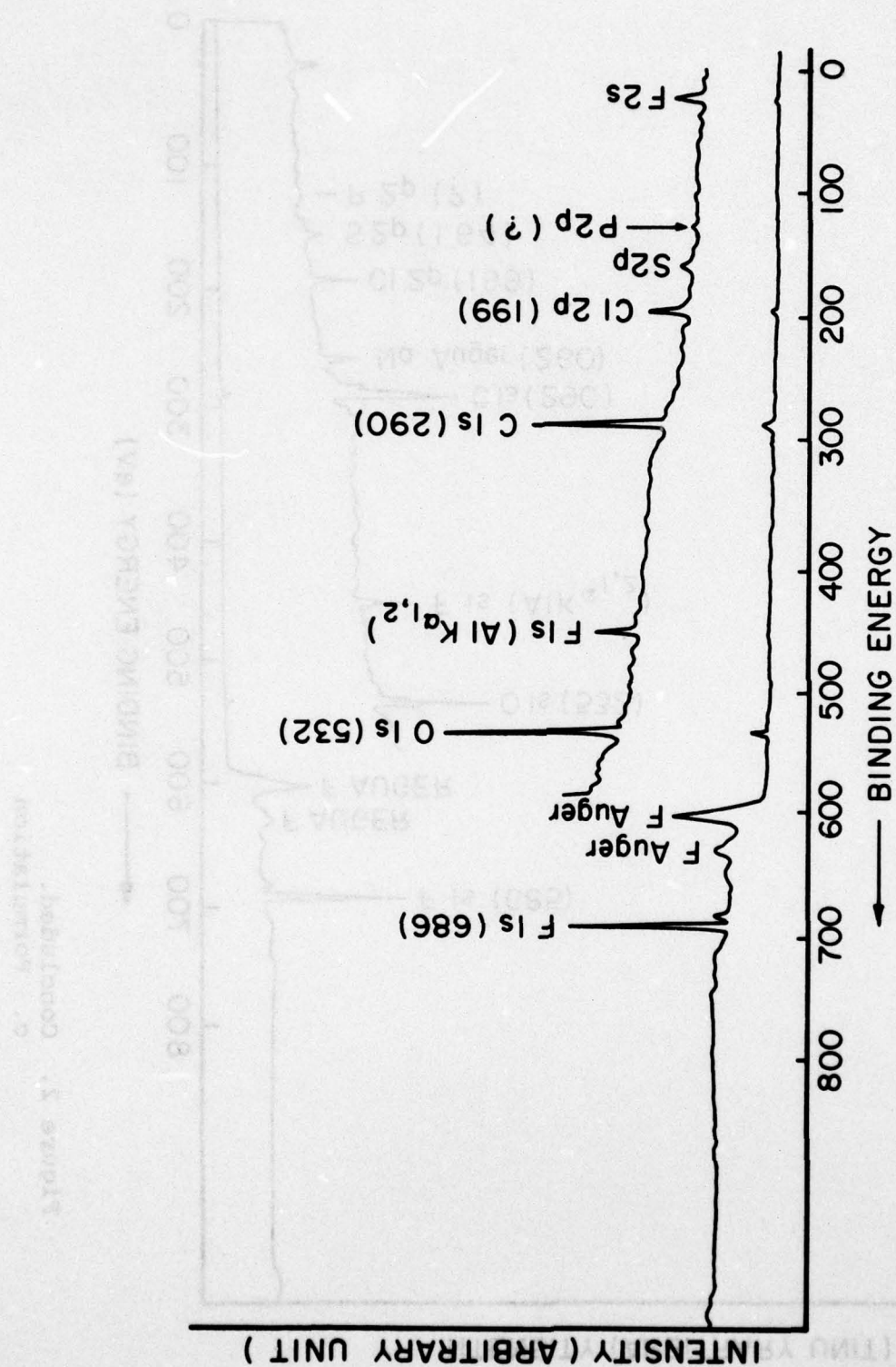
→ BINDING ENERGY

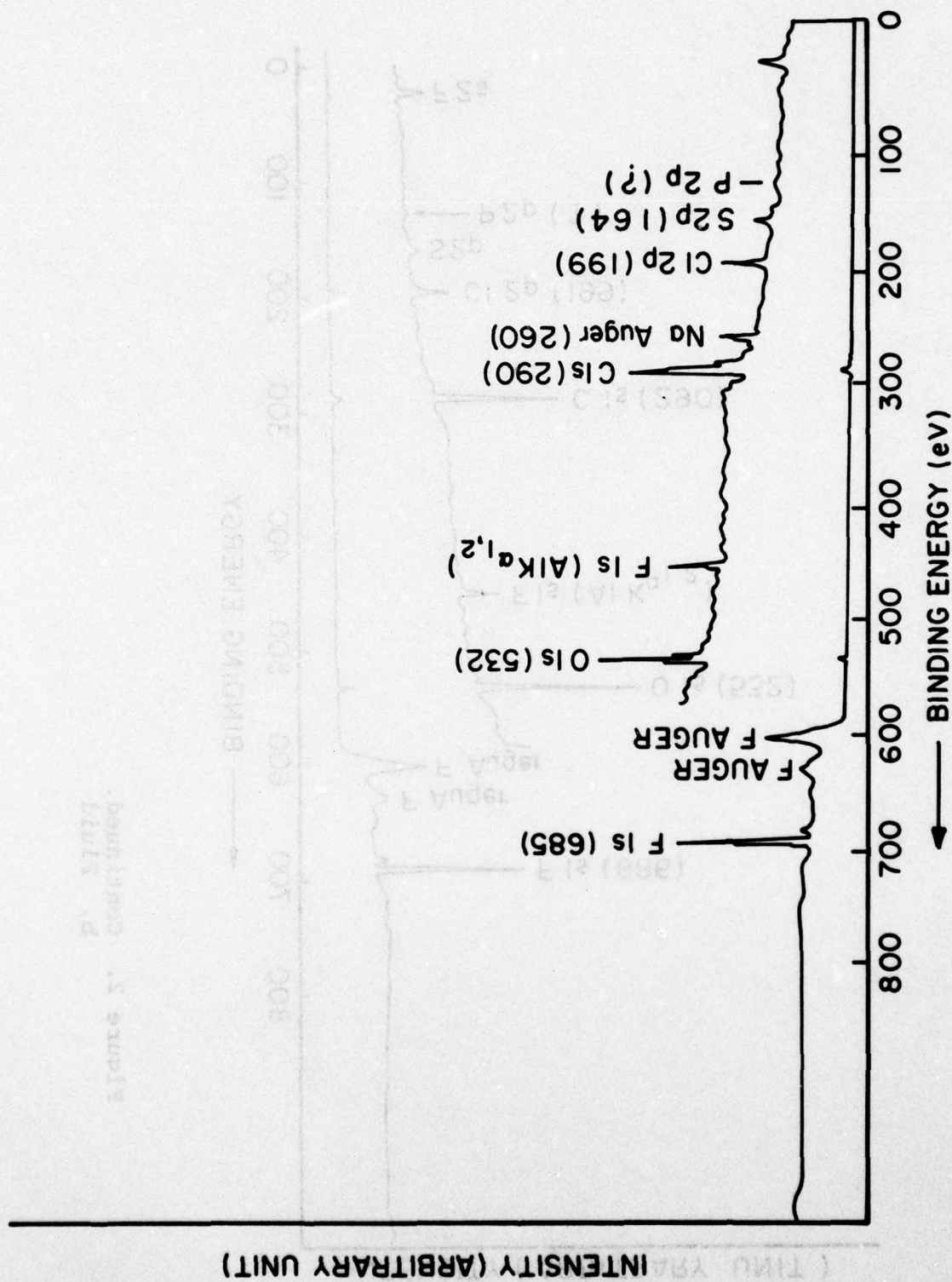INTENSITY (ARBITRARY UNIT)

Figure 2. Continued.
b. Fluid

Figure 2. Concluded.
c. Formulation

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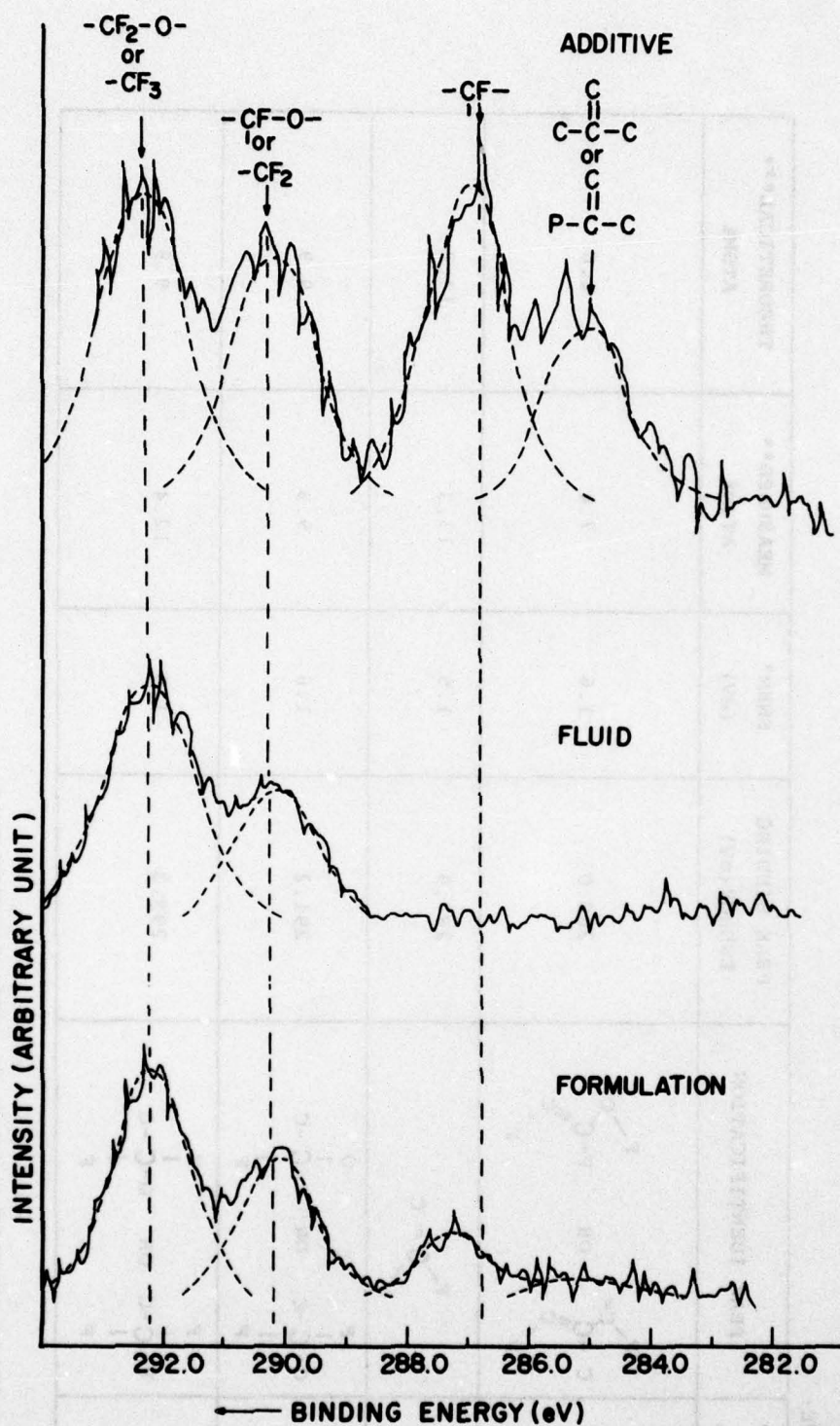
Figure 3. Carbon 1s Spectra of Additive, Fluid, and Formulation.

TABLE II

DATA OBTAINED FROM HIGH RESOLUTION, SINGLE-ELEMENT SCANS OF ADDITIVE, FLUID, AND FORMULATION

ADDITIVE:

ELEMENT & LEVEL	PEAK IDENTIFICATION	PEAK BINDING ENERGY(eV)	FWHM* (eV)	MEASURED** ATOM%	THEORETICAL*** ATOM%
C 1s	(structures)	286.0	1.6	7.4	6.6
C 1s	(structures)	287.9	1.5	13.3	13.2
C 1s	(structures)	291.2	1.6	9.9	9.9
C 1s	(structures)	293.3	1.6	12.4	9.9

*FULL-WIDTH-AT-HALF-MAXIMUM OF PHOTOELECTRON PEAK

**AS MEASURED FROM PEAK HEIGHTS

***AS CALCULATED FROM THE STRUCTURES GIVEN IN FIGURE 1

TABLE II (continued)

DATA OBTAINED FROM HIGH RESOLUTION, SINGLE-ELEMENT SCANS OF ADDITIVE, FLUID, AND FORMULATION

ADDITIVE:

ELEMENT & LEVEL	PEAK IDENTIFICATION	PEAK BINDING ENERGY (eV)	FWHM* (eV)	MEASURED** ATOM%	THEORETICAL*** ATOM%
O 1s		535.6	2.0	4.0	3.3
F 1s		689.2	2.3	51.5	56.0
P 2p		133.6	3.0	1.4	1.1

FLUID:

ELEMENT & LEVEL	PEAK IDENTIFICATION	PEAK BINDING ENERGY (eV)	FWHM* (eV)	MEASURED** ATOM%	THEORETICAL*** ATOM%
C 1s	O-C-C with F	291.0	1.6	13.4	10
C 1s	F-C-C OR C-C-O with F	293.1	1.7	24.5	20

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TABLE II (continued)

DATA OBTAINED FROM HIGH RESOLUTION, SINGLE-
ELEMENT SCANS OF ADDITIVE, FLUID, AND FORMULATION

FLUID:

ELEMENT & LEVEL	PEAK BINDING ENERGY (eV)	FWHM* (eV)	MEASURED ATOM%	THEORETICAL ATOM%
O 1s	534.3	1.9	10.7	10
F 1s	689.2	2.1	51.4	60

FORMULATION:

ELEMENT & LEVEL	PEAK BINDING ENERGY (eV)	FWHM* (eV)	MEASURED ATOM%
C 1s	288.4	1.4	6.1
C 1s	291.3	1.4	13.1
C 1s	293.4	1.6	21.2
O 1s	535.5	1.7	6.6
O 1s	531.7	1.9	2.6

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TABLE II (concluded)

DATA OBTAINED FROM HIGH RESOLUTION, SINGLE-
ELEMENT SCANS OF ADDITIVE, FLUID, AND FORMULATION

FORMULATION:

ELEMENT & LEVEL	PEAK BINDING ENERGY (eV)	FWHM* (eV)	MEASURED ATOM%
F 1s	689.2	2.0	47.9
P 2p	133.4	2.5	2.5

TABLE III

$\dfrac{C_{293}}{C_{291}}$ VALUES FOR FLUID, ADDITIVE, AND FORMULATION

Sample	$\dfrac{C_{293}}{C_{291}}$
Additive	1.25
Fluid	1.82
Formulation	1.62

As a first approximation, it seems reasonable to assume that a linear relationship exists between the ratio $\frac{C_{293}}{C_{291}}$ and the amount of additive present in the sample. Using this approximation, one would find the additive at a concentration of approximately 35 percent in the 1 percent formulation. Two reasons for these observations can be postulated. One possibility is that the formulation is not homogeneous, with selective precipitation occurring. Another possibility is that the additive and fluid have reacted to form new compounds. (A possible explanation for the C ls photopeak at 288 eV could be that it is due to an acid product).

Coupling the phosphorous data and the C ls binding energy data, both from Table II, supports the second reason. The formulation data shows an abnormally high phosphorous level. The theoretically expected value of phosphorous for the formulation is approximately .01 percent. The measured value was 2.5 percent; this is a factor of 2 higher than in the additive alone. This phosphorous data is difficult to explain in terms of a non-homogeneous mixture. It is more readily explained in terms of the additive reacting with the fluid.

The results in this section confirm that: a) the additive, the fluid, and the formulation appear to be slightly contaminated with chlorine and sulfur, b) the C-chemistry and elemental analysis observed by ESCA agrees well with that predicted from the known structures of the additive and the fluid, c) the ESCA Cls and P2p data on the formulation suggest that, (1) it is not homogeneous or (2) the additive chemistry has changed upon being mixed with the fluid, and d) the P2p binding energy of the additive was measured to be 133.6 eV and 133.4 eV for the formulation. This is characteristic of a phosphorous atom having an associated net positive charge.

SECTION IV
ANALYSIS OF COUPON SAMPLES

1.   ESCA OVERALL SCANS

ESCA overall scans were taken for each of the coupon samples submitted. Table IV gives the approximate atomic percent composition determined for each of the coupons. There are two samples of freshly polished metal coupons that were analyzed. In both cases, note the high levels of carbon and oxygen on the surface. There is also a trace of silicon found on both samples, probably from residual silicon of the polishing paper (SiC).

Three other metal coupons were analyzed. They are CS211I, CS220I, and CS225I. The first two were stressed in the 1 percent formulation. The third specimen was stressed in the fluid only. Determination of iron levels for samples CS211I and CS220I is complicated by the presence of a fluorine peak, due to a multiple excitation process, at the same binding energy as the most intense iron peak. The iron levels listed in Table IV are upper limits on the amount of iron present in these samples. There were regions on the CS220I sample which were corroded as well as regions which appeared protected from corrosion. The overall scan of a good (or protected) region is compared with a scan of a bad (or corroded) region in Figure 4. Note that there is an increased iron level and a decreased phosphorous level in the bad region.

For the unprotected regions of CS225I and CS220I, Table IV shows that there are increased iron and silicon levels on these samples as compared with the protected coupons.

2.   CHEMISTRY OF OXIDATION STATE STUDIES OF CS220I

High resolution scans of the carbon 1s, oxygen 1s, fluorine 1s, and phosphorous 2p peaks were taken for samples from good and bad regions of CS220I. These scans are comparable to those for the fluid and the additive. Table V presents the data obtained

TABLE IV

ATOM PERCENT COMPOSITION OF M-50 METAL COUPONS
(determined from ESCA all-element scans)

Sample	Iron	Fluorine	Oxygen	Nitrogen	Carbon	Chlorine	Phosphorous	Silicon	Sulfur	Gold*
1) Freshly polished (#605113)	4.2	--	27.2	1.8	60.9	1.1	<0.4	0.8	--	--
2) Freshly polished (#6102512)	2.4	--	16.0	1.0	75.0	1.2	<0.1	0.4	0.5	--
3) CS225I	6.5	7.2	26.2	1.7	54.7	0.3	<0.1	0.6	--	--
4) CS211I	<1.3	35.2	23.8	3.7	29.8	--	6.2	--	--	--
5) CS220I - good region	<1.2	26.7	30.0	5.2	28.2	1.0	7.6	--	--	--
6) CS220I - bad region	4.3	42.9	16.5	--	28.0	2.9	2.5	0.9	0.9	1.1

*From gold plated sample holder.

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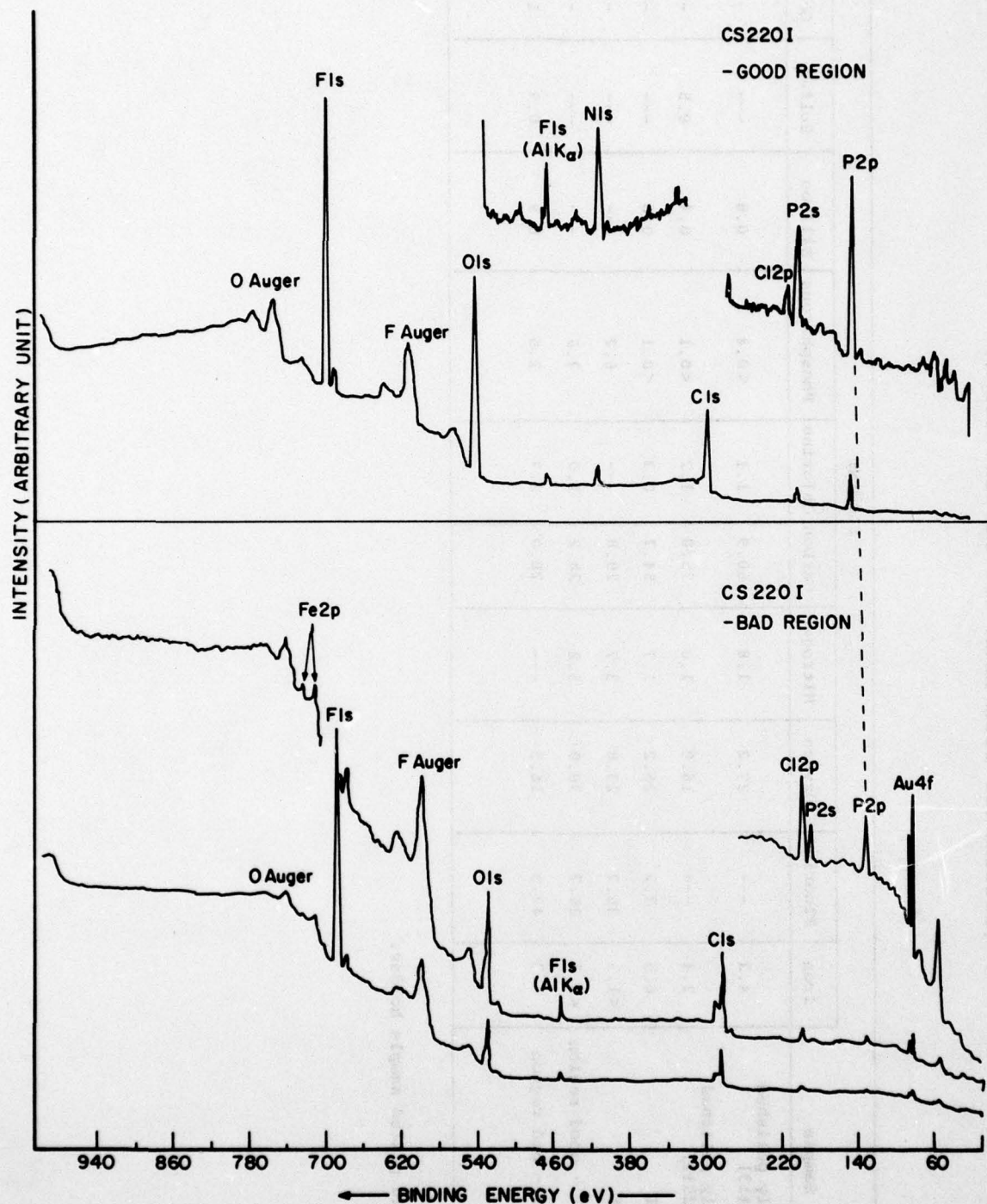
Figure 4.   ESCA Overall Spectra of Good and Bad Regions of CS220I.

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TABLE V

DATA OBTAINED FROM HIGH RESOLUTION,
SINGLE-ELEMENT SCANS OF CS220I

CS220I - good region:

Element & Level	Peak Binding Energy (eV)	FWHM (eV)	Measured* Atom %
C1s	285.0	1.9	25.1
C1s	287.2	2.0	4.9
C1s	288.4	1.9	8.7
C1s	290.9	1.9	2.6
C1s	293.5	1.6	2.8
O1s	531.7	1.8	20.0
O1s	533.3	1.8	12.9
F1s	688.7	2.1	16.3
P2p	133.6	2.0	6.7

CS220I - bad region:

Element & Level	Peak Binding Energy (eV)	FWHM (eV)	Measured* Atom %
C1s	285.0	2.0	14.7
C1s	286.7	1.7	4.5
C1s	288.0	1.7	3.5
C1s	291.0	1.6	7.3
C1s	293.5	1.7	12.2
O1s	531.0	1.8	9.5
O1s	532.8	2.2	5.0
O1s	535.4	1.9	4.5
F1s	689.1	2.1	39.0
P2p	(133.8)[+]	(2.0)[+]	(---)[+]

*The values in this column assume that the only elements in the sample are C, O, F, and P.

[+]Phosphorous was not measured at the same time as the other elements. For this reason the value for "Measured Atom %" was not included.

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from high resolution scans of the fluorine, oxygen, carbon, and phosphorous peaks for the two regions.

A F/P ratio of 51 for the additive can be calculated from the formula given in Figure 1. The measured ESCA value for the additive was found to be 37 (Table II). However, the F/P ratio on coupon samples which had good regions is on the order of 5, much less than expected if additive was simply bound to the surface. This data suggests that the compounds found on the iron substrate are some form of additive reaction products.

2.1   Chemistry Studies of Carbon

We noted a large amount of carbon on the iron substrate prior to contact with the additive/fluid formulation. We also noted that <u>no</u> carbon 1s existed at 285.0 eV in the additive or fluid. Thus, we conclude that this carbon is present on the steel prior to interaction with the additive/fluid formulation.

In Figure 5 the carbon 1s spectra of good and bad regions are compared with similar spectra from the additive and the formulation. One will immediately note the presence of a carbon 1s ESCA signal at ~285.0 eV on both the good and bad regions that is not present in the additive or fluid spectra. The possible sources of carbon-containing compounds with this particular binding energy are numerous. The exact nature and source of these carbon-containing compounds are uncertain at this time but is most likely due to C-C or C-H bonding. More work is needed to determine the nature of these compounds and whether they are evolved before, during, or after O-C testing.

For both the good and bad regions a shoulder can be noted to the left of the 285.0 eV peak. The width of the shoulder indicates that it is due to carbon from more than one chemical environment. This region of the spectrum is characteristic of C-F, C-O, or C=O type bonding. We have deconvoluted this shoulder into two peaks. An exact interpretation of this shoulder is a matter of speculation at this time. Analyses of standard compounds could perhaps elucidate the carbon bonding in this region.
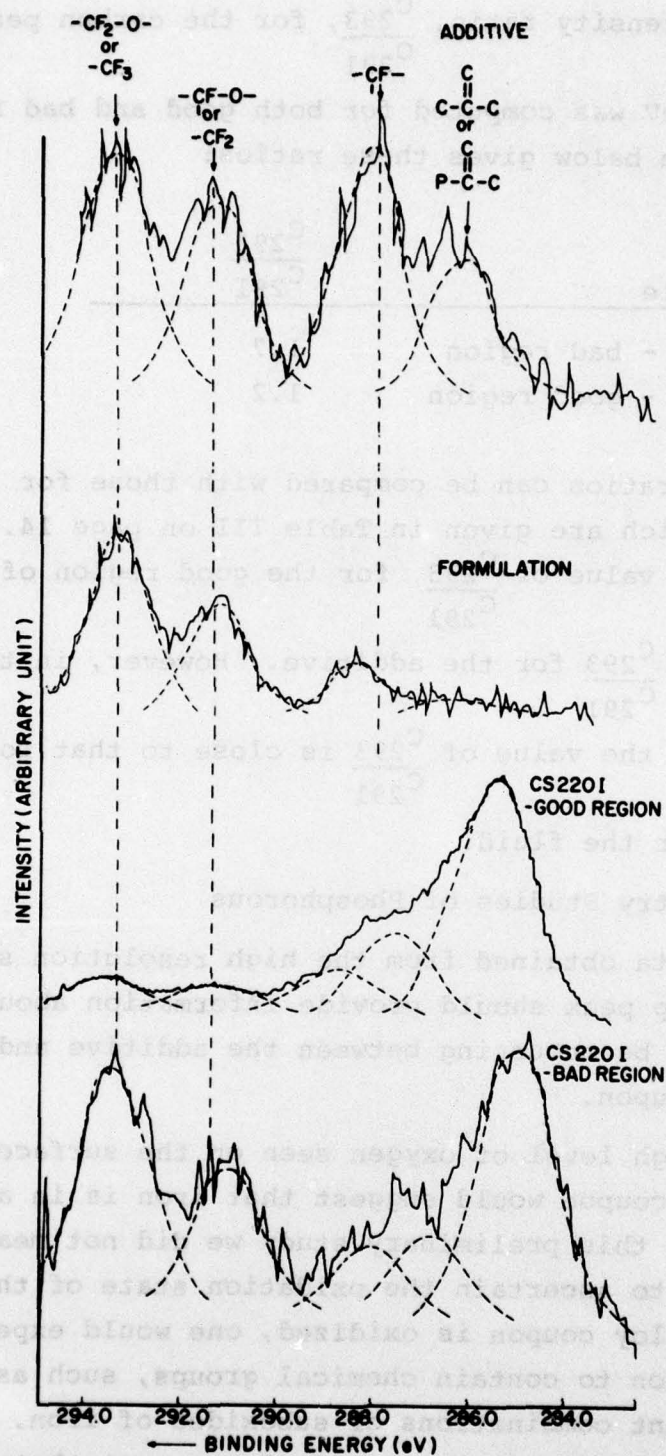
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Figure 5. Carbon 1s Spectra of Additive, 1 Percent Formulation, and Good and Bad Regions of CS220I.

The intensity ratio, $\dfrac{C_{293}}{C_{291}}$, for the carbon peaks at
~293 eV and ~291 eV was computed for both good and bad regions of
CS220I. The table below gives these ratios:

Sample	$\dfrac{C_{293}}{C_{291}}$
1) CS220I - bad region	1.7
2) CS220I - good region	1.2

These ratios can be compared with those for the additive and fluid which are given in Table III on page 14. It will be noted that the value of $\dfrac{C_{293}}{C_{291}}$ for the good region of CS220I is quite close to $\dfrac{C_{293}}{C_{291}}$ for the additive. However, in the bad region of CS220I, the value of $\dfrac{C_{293}}{C_{291}}$ is close to that for the formulation or for the fluid.

2.2   Chemistry Studies of Phosphorous

The data obtained from the high resolution scans of the phosphorous 2p peak should provide information about the bonding which may be occurring between the additive and the surface of the coupon.

The high level of oxygen seen on the surface of a freshly polished coupon would suggest that iron is in an oxidized state. (In this preliminary study we did not measure iron binding energies to ascertain the oxidation state of the iron). Since the iron alloy coupon is oxidized, one would expect the surface of the iron to contain chemical groups, such as Fe$\overset{O}{\frown}$Fe, F-OH, and different combinations of suboxides of iron. Since :P$<$ is a strong Lewis base, one would also expect that the bonding of the additive to the coupon would occur through the lone pair electron interaction of the phosphorous and the oxides

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or hydroxides of the coupon. Two possible mechanisms are: (1)
oxide formation, $Fe^{\hat{\delta}}-\hat{O}^{\delta+}\!:\!P\!\!<$ and (2) hydrogen bonding, $Fe-\bar{O}\hat{H}^{+\hat{\delta}}\!:\!P\!\!<$.
Both of these mechanisms would predict a net loss of electrons
from the phosphorous to the iron oxides or hydroxides. This
loss of electrons would cause an increase in the binding energy
of the phosphorous 2p level.

The binding energy of phosphorous (:P<) in triphenyl-
phosphine, :P$(C_6H_5)_3$, was measured by Hamer et al. to be 131.3 eV.[2]
The binding energy of phosphorous (-O:P<) in triphenylphosphine
oxide, O:P$(C_6H_5)_3$ was found by Zhavoron et al. to be 132.8.[3]
The phosphorous 2p binding energy for fluorinated triphenylphos-
phine has not been reported. We have measured the phosphorous 2p
binding energy in the additive and in the additive-fluid formula-
tion to be 133.4+0.2 eV. This binding energy as compared with
the literature values suggests that the phosphorous is highly
oxidized or that the lone pair is highly delocalized over the
fluorinated benzene ring.

Before placing too much confidence in the comparison
of the measured binding energies with the values found in the
literature, we need to measure more known compounds on the ESCA
AEI spectrometer at the University of Dayton. Experiments on
such compounds will elucidate the non-adjacent atomic potential
effects.

[2]A.D. Hamer, D.G. Tisley, and A.R. Walton, J. Chem. Soc.,
The Dalton Trans., 116 (1973).

[3]N.M. Zhavoron et al., Akad. Nauk SSR, Izv. Fiz., 36, 376 (1972).

SECTION V
CONCLUSIONS AND FURTHER SUGGESTIONS

The following is a list of conclusions concerning the ESCA experiments that have been performed to date.

1.   The process of polishing and cleaning produces coupon surfaces contaminated with oxygen (presumably oxides and hydroxides of iron).

2.   The process of polishing and cleaning produces surfaces that are contaminated with carbon-containing compounds having a C 1s binding energy of 285.0 eV. These compounds were also found after the washer had undergone O-C testing in the 1% formulation.

3.   The C 1s data for the O-C tested coupon samples indicate that the following carbon-containing groups are present on the surface: $-CF_3$ (or $-CF_2-O$), $-CF_2-$ (or $-CF-O-$), $= CF-$ (or $>C-O-$ or $= C=O$), and $>C-P<$.

4.   P 2p binding energies suggest that in all cases, phosphorous is in a trivalent state with a highly delocalized lone pair.

5.   Good regions of the coupons show increased levels of phosphorous and decreased levels of iron as compared to bad regions. This shows that a phosphorous compound is protecting the coupon.

6.   The phosphorous level on a good region of a coupon is ~5 times higher than in the additive alone. Also the F/P ratios are much lower for the good regions of the coupon than that predicted from the structure of the additive. This indicates that in the protective action afforded during the O-C test, the additive has changed chemistry.

We recommend that future experiments with ESCA could be conducted to investigate:

1.   The nature of the carbon-containing contamination on the surface of coupons.

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2.   The structure and thickness of oxides on the coupon surface.

3.   The effect of O-C testing on the chemistry of the perfluoro-polymer fluid and additive-fluid formulations:

a). in the presence of M-50 steel coupons;
b). without M-50 steel coupons.

4.   The stoichiometry of the phosphorous-containing protective film on metal coupons which are O-C tested in additive-fluid formulations.

AFML STANDARD POLISHING PROCEDURE
OF METALS FOR USE IN MICRO OXIDATION-CORROSION STUDIES

Metal coupons received from the manufacturer are first rinsed with technical grade acetone and/or petroleum ether to remove oil or other residues on the metal surfaces from the manufacturing, packing, or storing processes. All surfaces of the coupon are then polished using silicon carbide paper of various grit sizes (240, 320, 400, 500 grits are commonly used). A 500 grit paper (finest grit used) is used for the final polishing to give an approximation of a mirror finish to the coupon.

The actual polishing procedure for <u>new</u> coupons consists of the following steps:

1) Polish the outer and inner circumferences of the coupon first. The inner circumference polishing can be accomplished by wrapping a piece of 240 grit paper around a nail, slip coupon over the paper on the nail, and roll coupon on its outer circumference back and forth on a table covered with several layers of still paper towels (4-8 layers). Repeat with 500 grit paper. When the inner circumference is polished, the outer circumference is polished using same sized grit papers by rubbing the coupon in incremental steps around the coupon while holding the coupon firmly between the thumb and index finger.

2) The flat surfaces are now ready to be polished. Use a simple tool made from a #3 rubber stopper and a 3/8 inch diam. bolt as shown below to hold the coupon in position.

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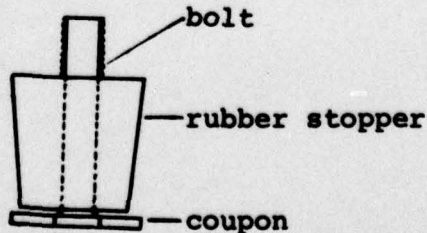
(It is important the bolt not be too tightly fit into the rubber stopper because that will make it difficult to adjust to the thickness of the coupon.) Rub the coupon over a piece of grit paper with several layers of toweling underneath the paper (to provide a more uniform surface) in a random pattern (preferably a figure eight). Use 240 grit paper first then the 500 grit paper to give a near mirror finish.

3) After the polishing process is complete, the coupon is rinsed with water to remove salts, with acetone to remove the water, then ultrasonically clean the coupon in petroleum ether to remove oils. Rinse the coupon with acetone then clean ultrasonically with acetone to remove organics.

For used coupons it seems easier to work up to the 500 grit paper using the 320 and 400 grit papers after the 240 grit paper. It is preferable to polish and clean the coupon as close to the actual weighing for a particular oxidation-corrosion test to insure a freshly polished surface with as little oxide formed as possible. It is important to use only one alloy per sheet of grit paper in order to prevent contamination from different metals.

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